T1-2494

ANSTIA 4090

WADC TECHNICAL REPORT 52-100

AD 0004090

DO NOT FIGUREY

RETRY 19
TECHTON 100 100 101
COMPAND 100 100 100
WOOSE3

CATALOGE AS AD

IMPROVEMENT OF JET ENGINE DESCALING PROCEDURE

G. M. BRYAN
NORTHROP AIRCRAFT, INC.

Because of our limited supply you are reque this copy as soon as it has served your pit may be made available to others for your cooperation will be appreciated.

RAKEL S

WRIGHT AIR DEVELOPMENT CENTER

20020326068

Statement A Approved for Public Release

AF-WF-(8)-0-6 MAR 83 456

NOTICES

When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related Government procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact—that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data, is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

The information furnished herewith is made available for study upon the understanding that the Government's proprietary interests in and relating thereto shall not be impaired. It is desired that the Judge Advocate (WCJ), Wright Air Development Center, Wright-Patterson Air Force Base, Ohio, be promptly notified of any apparent conflict between the Government's proprietary interests and those of others.

0000000000

IMPROVEMENT OF JET ENGINE DESCALING PROCEDURE

G. M. Bryan Northrop Aircraft, Inc.

August 1952

Materials' Laboratory
Contract No. AF 33(038)-23310
RDO No. 611-14

Wright Air Development Center Air Research and Development Command United States Air Force Wright-Patterson Air Force Base, Ohio

FOREWORD

This report is a summary of research on the "Improvement of Jet Engine Descaling Procedure" under Contract AF 33(038)-23310. (This work was initiated at Northrop Aircraft, Inc., as a sub-project under Research and Development Order No. 611-14, "Cleaning Compounds and Techniques.") The contract was administered under the direction of the Materials Laboratory, Directorate of Research, Wright Air Development Center, with Capt Harry J. Packer acting as project engineer.

The author wishes to thank the personnel of the Materials Laboratory for their help and cooperation. Acknowledgement is also due to the officers, enlisted men and civilian personnel at Edwards Air Force Base, Tinker Air Force Base, and Norton Air Force Base for the interested and friendly assistance which they afforded the author in every phase of the program. And finally he must offer his gratitude and best wishes to all of the manufacturers of metal cleaning compounds who worked with Northrop Aircraft, Inc., in the development and evaluation of descaling formulations.

ABSTRACT

X-ray diffraction analysis of the Inconel scale on combustion tube inner liners revealed that it is composed of nickel oxide, the major component, and lesser amounts of chromium oxide and iron oxide. Metallographic examination demonstrated that heavy scale is associated with precipitation, possibly carbides at the grain boundaries, a fact which can explain intergranular corrosion of scaled Inconel in acid solutions.

Incomel scale could not be taken off in neutral or alkaline solvents but several acid solutions were found which remove most of the scale without serious-ly attacking the base metal. Oxidizing pretreatments, particularly with the alkaline permanganate solution in current use by the Air Force, were shown to promote efficient acid pickling.

Physical tests of Inconel specimens descaled with the nitric acid-ferric chloride solution revealed that high temperature pickling (160°F) caused a severe loss in tensile strength whereas room temperature pickling caused no appreciable loss in tensile strength.

A full scale test of the nitric acid-ferric chloride solution was performed at Norton Air Force Base, San Bernardino, and satisfactory results were achieved.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDING GENERAL:

M. E. SORTE

Colonel, USAF

Chief, Materials Laboratory

Directorate of Research

TABLE OF CONTENTS

		PAGE
Section I	Survey of Current Jet Engine Cleaning Methods	1
Section II	The Origin and Nature of Inconel Scale	2
Section III	Development of Neutral and Alkaline Descaling Systems	5
Section IV	Development of Acidic Descaling Systems	6
Section V	Descaling Materials and Methods Developed by Vendors	14
Section VI	Conclusions and Recommendations	16
Bibliography		17
Appendix I	Jet Engine Cleaning at Tinker Air Force Base, Oklahoma City, Oklahoma	18
Appendix II	Jet Engine Cleaning at Norton Air Force Base, San Bernardino, California	쇰
Appendix III	Jet Engine Cleaning at Edwards Air Force Base, Muroc, California	27
Appendix IV	Thermal Analysis of Turbo Jet Combustion Zone	29
Appendix V	Heat-Resistant Alloys for Use in Jet Engines	30
Appendix VI	X-Ray Diffraction Analysis of Heat Scale Formed on Inconel Liner	32
Appendix VII	Selection of Samples and Preparation of Specimens	33
Appendix VIII	Alkaline and Neutral Solvents Tested as Inconel Descaling Solutions	34
Appendix IX	The Effect of Pure Acids in Various Concentrations Upon Inconel Scale	3 5
Appendix X	Acid Formulations Containing Reinforcing Agents	3 6
Appendix XI	A. The Effect of Variation in Nitric Acid Strength and Ferric Chloride Concentration Upon the Descal- ing Activity of the NF Acid	
	B. The Effect of Sodium Chloride as an Activator	38
Appendix XII	The Effect of Repso MF and Dibutylthiourea in Certain Descaling Acids	3 9
Appendix XIII	The Effect of Various Oxidizing Pretreatments in the Descaling of Stainless Steel with the NF Solution	40
Appendix XIV	The Effect of Various Descaling Procedures Upon the Tensile Strength of Inconel	41
WADC TR 52-100	iv	

		PAGE
Appendix XV	The Analysis of the NF Solution	42
Appendix XVI	Questionnaire Survey Introductory Letter Questionnaire Distribution List Recommended Scale Removal Methods	44 44 45 47 50
Appendix XVII	The Vendor Program Letter of Sollicitation Vendor Participants	51 51 53

INTRODUCTION

The purpose of this project, initiated under Contract No. AF 33(038)-23310, was the development of improved processes and techniques for cleaning turbo jet or gas turbine engine parts. Various reasons why the present cleaning methods prescribed in T.O. 02B-1-13 "Cleaning of Turbo Jet or Gas Turbine Engine Parts" are unsatisfactory for certain engine components are presented in PR No. 92954. Briefly, the objections raised against the present process, which utilizes an oxidizing pretreatment in hot alkaline potassium permanganate followed by a brief dip in an acid pickling bath, are:

- 1. The action of the permanganate bath is rather slow and constitutes a bottleneck in production. Furthermore, potassium permanganate is relatively expensive and apt to be in very short supply in time of emergency.
- 2. The use of acid materials to remove the oxidized scale is likely to result in the corrosion of engine components made up of dissimilar metals or of metals which may have been rendered peculiarly sensitive to intergranular attack by the high temperature of previous service operation.

The suggestion was made in PR No. 92954 that in view of the difficulties encountered in the use of acid pickling systems, an effort be made to develop a wholly alkaline cleaning system employing such well known chemical expedients as oxidation-reduction, catalysis, preferential absorption, and the formulation of complexes. The view was expressed that such a system would not effect the severe intergranular corrosion which often occurs in acid solutions.

SECTION I - SURVEY OF CURRENT JET ENGINE CLEANING METHODS

The attainment of near sonic velocities by modern military airplanes is largely the result of the tremendous thrust generated by their turbo jet engines. The high operating temperatures of these power plants and the great stresses induced in the engine components necessitates frequent and thorough inspection to insure safe and efficient operation. Engine cleaning therefore assumes an important role in the maintenance program as a pre-inspection procedure intended to facilitate the discovery of minute cracks and dimensional changes before these incipient failures have resulted in major engine damage.

During the survey of current jet engine cleaning methods conducted as the preliminary phase of this work, the author visited three Air Force bases where jet engines are overhauled: Tinker Air Force Base, Oklahoma City, Oklahoma; Norton Air Force Base, San Bernardino, California; and Edwards Air Force Base, Muroc, California.

Tinker Air Force Base and Norton Air Force Base are major overhaul depots and are equipped to disassemble, clean, and rework over twenty jet engines per day while Edwards Air Force Base, being essentially an experimental depot, is equipped to handle only the maintenance of the relatively small number of engines associated with the development program.

The engine cleaning at Muroc is keyed to simplicity and "Serviceability" is the watchword of the maintenance program. At the two major depots, however, the appearance of the engine components is granted equal consideration with serviceability and extensive efforts are made to turn out overhauled engines which are as bright and shiny as brand new power plants.

Norton Field and Tinker Field differ in their jet engine maintenance programs chiefly in the fact that the San Bernardino Base is the more modern and has incorporated several noteworthy advancements which were developed in an effort to improve the general operation pioneered at Tinker Field. These improvements are:

- (1) A better arrangement of cleaning tanks coupled with a superior overhead monorail system.
- (2) Cleaning tanks which are adaptable to a wide variety of cleaning materials.
- (3) Vapor Blast and Grit Blast equipment incorporated as integral parts of the jet engine cleaning system.

Detailed descriptions of the jet engine cleaning operations at these three Air Force Bases are presented in Appendixes I, II, and III.

SECTION II - THE ORIGIN AND NATURE OF INCONEL SCALE

Since the scale is undoubtedly of an oxide character because of the high temperature oxidizing conditions under which it is formed, some idea of the temperatures developed in various parts of the engine where scaling occurs might be useful in gaging the magnitude and severity of the problem. Appendix IV presents some of the engine "hot" parts in sequence and lists the average operating temperature of each part as well as the transient maximum temperatures which may be attained as the result of direct flame impingement. A list of the metals which have been used or have been proposed for use in jet engines is presented in Appendix V.

Vapor blast is frequently used to remove the scale from nozzle diaphragms and turbine wheels, neither of which contain Inconel. Outer combustion chambers, which are generally fabricated from a stainless steel such as Type 347, are not subject to severe intergranular attack in the ferric sulphate-hydrofluoric acid solution because of the relatively low temperature of these parts during engine operation. The inner combustion chambers, or flame tubes, and the transition liners are made of Inconel and because of the high temperatures of these parts during engine operation, particularly under transient conditions, are subject to severe intergranular attack in certain acid solutions. In view of the magnitude of the Inconel pickling problem, and because of the fact that the greater portion of the scrap material furnished to this laboratory by the USAF was composed of Inconel flame tubes and transition liners, the major research effort at Northrop Aircraft, Inc. was concerned with the removal of Inconel scale.

It is not unreasonable to assume that the scale which forms upon Inconel components during the operation of a jet engine is a mixture of the oxides of the metals which comprise the alloy. The relative proportions of the various oxides in the scale may approach but need not correspond exactly with the percentages of the parent metals in the alloy. Despite the fact that some work has been done towards the elucidation of the nature of the oxide scales which form upon certain stainless steels in various environments no investigation has been reported in the literature concerning the nature of Inconel scale. Yamaguchi, Nakayoma, and Katsurail made electron diffraction measurements of the scale formed on 18 Cr, 4 Ni, and on 19 Cr, 9 Ni stainless steels by water vapor at high temperatures. Their data indicate that the scale could be a solid solution of nickel and iron chromates. McCullough, Fontana, and Beck 2 conducted electron diffraction studies on the scale formed on types 304 and 430 stainless steels and found that the initial scale formed was of the nature of an iron oxide, chromic oxide spinel.

The low solubility rate of spinels in acid is characteristic of the oxide combinations which comprise these minerals and the behavior of Inconel scale is very similar in this respect. It is not unlikely that the combination of the metal oxides which comprise the scale upon Inconel participates somewhat in the spinel structure and that this arrangement accounts for its refractory character.

Because no information concerning the nature of Inconel scale could be found in the literature an X ray diffraction study of the scale was made. With considerable effort a small quantity of the extremely refractory scale was obtained by mechanical abrasion of the surface of a flame tube and then subjected to the well known technique of powder crystallography. Two outside independent laboratories conducted X ray analyses of this material. Dr. T. Hall of Stanford University found that the scale was composed principally of nickel oxide (NiO) with lesser amounts of chromic and ferric oxides (Cr2O3 and Fe2O3). Details of his analysis are not available.

The Metal Control Laboratories of Huntington Park, California, obtained the same results with a similar sample of Inconel scale. These analysts found some indication that on iron nitride (Fe2N) was also present in the scale. Details of their analysis is presented in Appendix VI.

All subsequent work towards the development of a suitable descaling technique, both in the Northrop Aircraft, Inc. laboratory and in the laboratories of cooperating vendors, was based upon the knowledge that Inconel scale is largely a mixture of metal oxides with nickel oxide (NiO) the major component.

Since the formation of the oxide scale is undoubtedly influenced by the temperature and the composition of the hot combustion gases, the color, depth, and refractory character of the scale might be expected to depend upon these variables. In fact the differences in the characteristics of the scale are so great from one area of a flame tube to another that faulty interpretations of test cleaning data can easily be made unless this variation is taken into account. These differences in the characteristics of the scale itself are matched by greater but more subtle differences in the susceptibility of the base metal to intergranular corrosion in acid solutions.

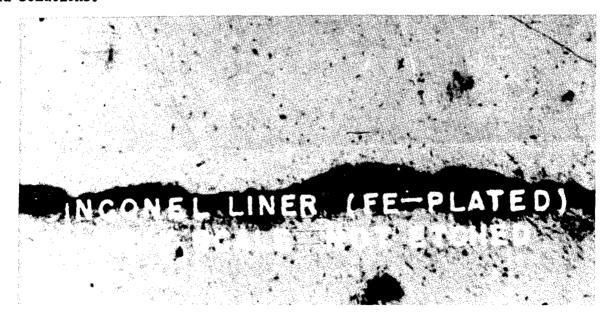


Figure 1. HEAVY INCONEL SCALE - Magnification 500X

Figure 1. illustrates the heavy oxide scale formed on an Inconel flame tube during a relatively short service period estimated at 150 hours. The scale is covered by a layer of iron which was deposited electrolytically on the specimen in order to preserve the scale during mounting and polishing. The inside surface of the liner was more heavily scaled than the outside surface. In the heavily scaled areas of the liner, intergranular oxidation penetration was observed to have taken place to a maximum depth of 0.0004 inches. Precipitation at grain boundaries, possibly carbides, is greater under heavily scaled areas than under lightly scaled areas.

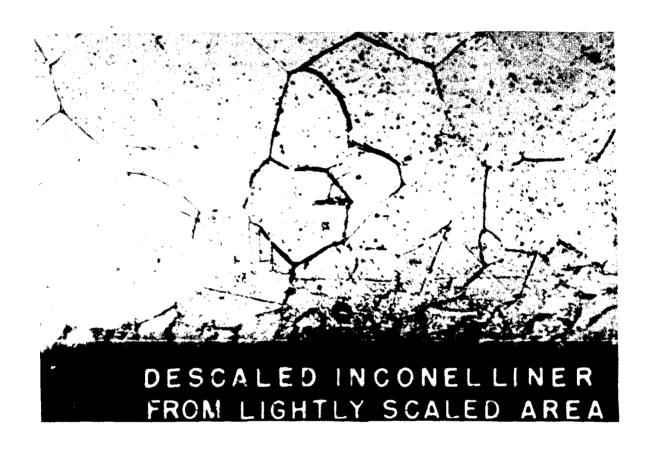


Figure 2. DESCALED INCONEL FROM LIGHTLY SCALED AREA - Magnification 500X

Figure 2. shows a section of a descaled Inconel liner. This specimen, cut from a lightly scaled area, was cleaned by a one hour soak at room temperature in a 25% nitric acid, 2.3% ferric chloride hexahydrate solution. Only slight damage to the base metal can be noted in the form of intergranular corrosion along the grain boundaries close to the metal surface.

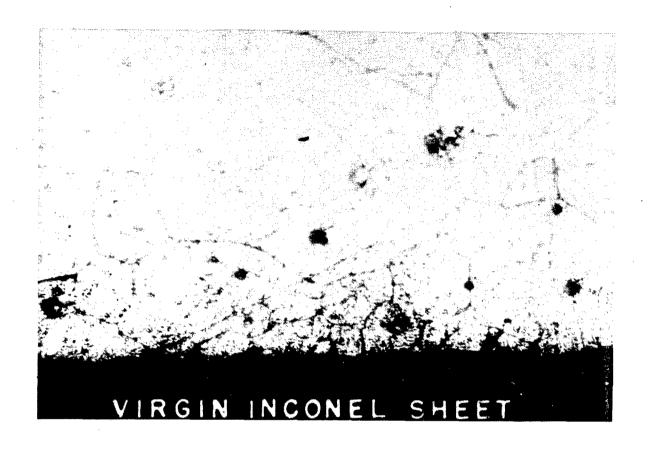


Figure 3. VIRGIN INCONEL SHEET - Magnification 500X

Figure 3. shows a section of virgin Inconel sheet for comparison. It can be observed that precipitation at the grain boundaries of the lightly scaled Inconel is no more predominant than in virgin sheet stock.

The occurrence of precipitation under the heaviest Inconel scale is the root of the descaling problem inasmuch as conditions which cause the development of the most refractory scale renders the base metal susceptible to corrosion in the acids which attack the oxide coating. This lowered resistance to intergranular attack might be attributed to the formation of carbides at the grain boundaries which deplete grain surfaces of the elements (principally chromium) which are necessary for acid resistance.

SECTION III - DEVELOPMENT OF NEUTRAL AND ALKALINE DESCALING SYSTEMS

In the investigation of alkaline and neutral solvents as well as in the investigation of acidic solvents, Inconel samples were selected at random from a stockpile of cut specimens but each solution tested was allowed to act upon all three types of scale - heavy, medium and light. These experiments, which can be considered as screening tests, were followed by further work in which a more rigorous method of sample selection was used. Thus, the one effective solution developed at Northrop Aircraft, Inc. the NF formula, and all promising vendors solutions were given as fair and objective evaluation as was possible. The details of this rigorous method of sample selection, by which the NF solution and all promising vendors solutions were evaluated, is presented in Appendix VII.

The initial attempts to remove scale from Inconel were made in alkaline and neutral solvents following the line of research suggested in PR No. 92954. The results were negative. A list of the solutions used and their effects upon the scale are presented in Appendix VIII. The fact that Inconel scale is insoluble even in oxidizing alkaline solvents is understandable inasmuch as the major scale constituent, nickel oxide, NiO, is not amphoteric. Although it is not unreasonable to suppose that some chromium oxide, Cr2O3, might be oxidized in an alkaline oxidizing solvent, no evidence of soluble chromate formation was observed in any of the experiments. After these explanatory tests failed to indicate that Inconel scale might be removed in neutral or alkaline solutions, work along the line suggested in PR No. 92954 was abandoned in favor of the investigation of acid systems.

SECTION IV - DEVELOPMENT OF ACIDIC DESCALING SYSTEMS

The first acidic pickling agents tested were pure acids and acids of various concentrations in water. As the results presented in Appendix IX, indicate, acids alone are not very effective as pickling agents for the removal of Inconel scale. Some reinforcement of the acid, either with a powerful oxidizing agent, a strong complexing reagent, or a catalytic salt is apparently necessary to promote the attack of the acid upon the refractory mixture of oxides which constitutes the scale.

Because of the fact that the oxidizing acid, nitric acid, was the most effective pickling agent of the straight acids tested, it was used as the substrate in most of the experiments with reinforcing agents. The results of these experiments involving the addition of various reinforcing agents to nitric acid is presented in Appendix X.

It is evident that the addition of a powerful oxidizing reagent such as potassium chlorate to the nitric acid solution creates an effective descaling medium. The great reactivity of potassium chlorate in acid solutions relative to its lower activity in neutral or basic solution can be predicted from the half cell reactions 4:

$$C10_3 + 6H + 5E = 1/2 Cl_2 + 3H_{20}$$
 Eo = 1.43 volts
 $C10_3 + 3H_{20} + 6E = C1 + 6OH$ Eo = .62 volts

It is obvious from the standard electrode potentials of the chlorate reduction half cells that the presence of hydrogen ion is necessary for the full utilization of the oxidizing strength of potassium chlorate.

The relatively short life of the nitric acid-potassium chlorate solution results from the decomposition of chloric acid and the subsequent loss of chlorine dioxide from the solution 5:

$$2HC1O_3 = HC1O_2 + HC1O_4$$

 $-HC1O_2 + HC1O_3 = 2C1O_2 + H_2O$

The very powerful complexing reagent, hydrofluoric acid, also creates an efficient descaling solution when added to nitric acid. Although this combination is widely used to remove heat treat scale from newly fabricated Inconel parts, its application to the removal of service scale from Inconel flame tubes and transition liners causes intergranular corrosion because of the sensitized condition of the metal. The effect of various metallic ions such as Fe the Nitro Sn. etc. in aug-

menting the descaling activity of nitric acid is a well recognized phenomenon. It is possible that a depolarizing action is responsible for this effect, or, considering the oxidizing action of the nitric acid, it is possible that the metallic ion participates catalytically in a low activation energy reaction.

The 25% nitric acid - 2.3% ferric chloride hexahydrate solution (2.55 molar nitric acid - 0.1 molar ferric chloride) was the most effective and least corrosive of the reinforced acids. This formulation, which will be designated as the NF solution, removes all but the heaviest scale at room temperature and causes very little intergranular corrosion. The hot solution is very aggressive, however, and promotes severe intergranular corrosion in the process of removing very heavy scale. The intergranular corrosion produced in an Inconel specimen by the hot NF solution is illustrated in Figure 4.



Figure 4. INTERGRANULAR ATTACK CAUSED BY HOT NF SOLUTION - Magnification 500X

In an experiment where the concentration of ferric chloride was varied from 0.001 molar to 0.100 molar in 25% nitric acid, the descaling activity of the NF acid appeared to be independent of the ferric chloride concentration. A reduction of the nitric acid concentration from 25% to 10% caused an appreciable reduction in descaling activity. The substitution of sodium chloride for ferric chloride did not diminish the activity of the solution. The concentrations of nitric acid, ferric chloride, and sodium chloride used in these experiments is presented in Appendix XI.

A test of the cationic wetting agent Repso MF (Refined Products Co.) as an additive to the NF solution demonstrated that although the initial activity of the NF solution was enhanced by the wetting agents, no permanent improvement was effected. The addition of 0.25% Repso MF and 0.5% dibutylthiourea to 20% hydrochloric acid greatly enhances the descaling activity of the acid and reduces the inter-

granular corrosion. Because of the fume problem, however, the hydrochloric acid formulation was not considered applicable to descaling in present USAF jet engine overhaul facilities. The results of these experiments are tabulated in Appendix XII.

Because an oxidizing pretreatment with alkaline potassium permanganate is a standard procedure in the present USAF process for descaling heat resistant alloys, it became a matter of interest to investigate the effect of various oxidizing pretreatments upon the descaling efficiency of the NF solution. In the first experiment designed to evaluate the effect of various oxidizing pretreatments upon the performance of the NF solution specimens of stainless steel cut from an outer combustion chamber of a J-33 engine were given a "laboratory" scale in an electric furnace (because there was no scale on the part as received) and then treated with the NF solution after various oxidizing pretreatments. The results of this test, which are presented in Appendix XIII showed that the alkaline permanganate solution (TO-02-1A-13) was superior to the other oxidizing agents tested. Alkaline potassium persulfate solution was effective over a much longer period (12 hours) as compared to 2 hours for alkaline permanganate but its action is much too slow to permit its use in the oxidizing pretreatment of jet engine components. Because of the rapid decomposition of persulfate at higher temperatures, the potassium persulfate solution must be used at ambient temperature, a disadvantage not shared by alkaline potassium permanganate.

The effects of several oxidizing pretreatments in the descaling of turbine blades were also evaluated. Descaling tests were conducted with four different acids including the NF solution following three oxidizing pretreatments. The results of this experiment, which are illustrated in Figure 5, demonstrates the fact that a very powerful acid such as 25% nitric acid - 3% hydrofluoric acid mixture obscures the relative effects of various oxidizing pretreatments by performing a thorough job of oxide removal in every case. With weaker and less corrosive acids, however, the relative superiority of the alkaline permanganate solution is clearly evident. It is interesting to note that several turbine blades could be almost entirely freed of scale and other surface contaminants by the action of the alkaline permanganate without subsequent acid treatment.

Figure 5 TURBINE BUCKETS CLEANED WITH VARIOUS OXIDIZING PRETREATMENTS

In an experiment similar to the one just described it was demonstrated that the alkaline permanganate solution was superior as an oxidizing pretreatment for Inconel. The efficacy of this pretreatment in promoting the removal of Inconel scale by the NF solution is illustrated in Figure 6. The six split columns of test specimens were cut from the side section of an Inconel flame tube. The control specimens on the left side of each column were cleaned in the NF solution at room temperature without any pretreatment while the pieces on the right had the benefit of a two hour oxidizing pretreatment in the alkaline permanganate solution at 190°F.

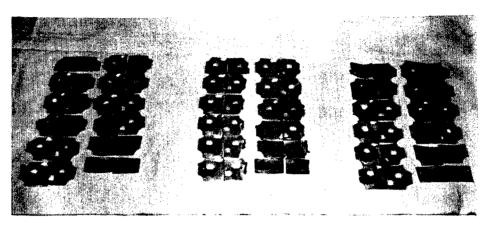


Figure 6. THE EFFECT OF THE ALKALINE PERMANAGANATE OXIDIZING PRETREATMENT IN THE DESCALING OF INCONEL

It can hardly be denied, in view of the results observed in the descaling of the three alloys which probably constitute the major portion of the "hot" metal in a jet engine, that an oxidizing pretreatment is very beneficial in the removal of scale. The benefits which accrue to the usage of such a process grow as the strength and corrosiveness of the subsequent acid pickle diminishes. Thus a two hour soak in hot alkaline permanganate allows Inconel flame tubes and transition liners to be almost thoroughly descaled in the NF solution at room temperature where the amount of intergranular corrosion is slight.

It was hoped at the outset of the Jet Engine Descaling Project at Northrop Aircraft, Inc. that a substitute for the alkaline potassium permanganate might be found. Despite its cost and slow action relative to the subsequent acid pickle, it was found to be the strongest and fastest alkaline oxidizing pretreatment available and because of this fact it will probably continue to be a part of the standard USAF jet engine cleaning procedure.

Inasmuch as "wet" oxidizing pretreatments proved to be very effective in promoting scale removal in acid solution it was decided that the fused salt pretreatments should also be investigated. Accordingly the following experiments were conducted with the friendly assistance of three companies which have molten salt descaling facilities.

At Ryan Aircraft Company, San Diego, several flame tubes were immersed for twenty minutes in the Virgo bath, a mixture of molten sodium hydroxide and sodium nitrate, and then pickled in a nitric acid-hydrofluoric acid solution. All of the scale was removed by this treatment but some evidence of intergranular corrosion was observed in certain areas. A solution heat treat at 1800°F. for five minutes prior to the Virgo treatment reduced the amount of corrosion which occurs in the acid pickle.

The Solar Aircraft Company of San Diego provided facilities for testing the Du Pont Sodium Hydride Process. A J-47 flame tube was immersed for twenty minutes in the reducing salt bath, molten sodium hydroxide containing 1/2 to 1 1/2% by weight of sodium hydride. Following the immersion in the salt bath, the flame tube was quenched in water and then pickled for five minutes in each of the two acid baths, 5% sulfuric acid and 25% nitric acid. This flame tube descaled with the help of the Du Pont Sodium Hydride Process was the most perfectly cleaned specimen ever seen by the Northrop laboratory staff. It is shown on the right hand side of Figure 7.

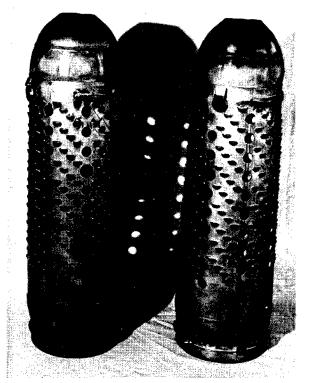


Figure 7.

R.H. FLAME TUBE CLEANED BY SCDIUM HYDRIDE PROCESS

L. H. FLAME TUBE CLEANED BY WYANDOTTE NITRO MF BATH

The objection of the USAF to fused salt baths was clearly substantiated by an experiment performed for us by the Electrochemicals Department of the Du Pont Corporation. Sections of a J-47 flame tube returned to this laboratory after the Sodium Hydride treatment were perfectly clean and apparently salt free when received but subsequently grew "whiskers" at the seams during an observation period of several weeks duration. Figure 8 illustrates the growth of alkaline salt material at the faying surfaces after the Du Pont molten salt treatment.

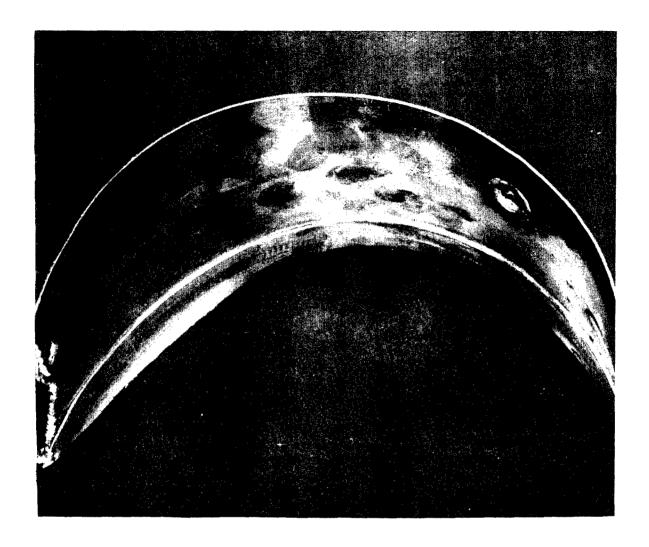


Figure 8. SECTION OF FLAME TUBE CLEANED BY SODIUM HYDRIDE PROCESS

Fused salt baths require considerable space and rather elaborate equipment and their operation is attended by a considerable personnel hazard. These difficulties in conjunction with the virtual impossibility of removing salt residue from crevices and lap joints outweigh their advantages from a descaling point of view and render them unsuitable for jet engine cleaning. The NF solution was evaluated with Inconel specimens selected and prepared according to the method outlined in Appendix VII. It was found to remove practically all of the scale at room temperature if the acid pickling were preceded by the conventional oxidizing pretreatment in hot alkaline potassium permanganate solution. The efficacy of the NF solution against the scale formed upon certain stainless steels in oxidizing atmospheres is inferior to other acid formulations, notably to those containing hydrofluoric acid. It is believed, however, that the oxidizing pretreatment with alkaline permanganate will allow the NF solution to compete favorably with the

stronger and usually more corrosive acids.

The NF solution produces but little intergranular attack at room temperature but is very corrosive at higher temperatures. (See Figures 2 and 4.) Final and conclusive evidence concerning the variation of intergranular corrosion in the NF solution with temperature and of the beneficial effect of an oxidizing pretreatment was obtained by the determination of the tensile strength of Inconel treated by various descaling procedures. Tensile test coupons were machined from virgin Inconel sheet stock and from the metal which formed the base of a J-47 flame tube. The effect of the various descaling procedures upon the tensile strength of the metal is presented in Appendix XIV.

It is very interesting to note that the tensile strength of the virgin Inconel sheet stock was unaffected by any of the descaling procedures. The flame tube metal, on the other hand, suffered a considerable loss in tensile strength when it was pickled in the hot acid solution. The difference in acid resistance between virgin Inconel and the metal which has been exposed to the high temperatures of jet engine operation could hardly be better illustrated than by a comparison of the effect of hot NF acid upon the respective tensile properties.

The room temperature application of the NF acid to the descaling of flame tube Inconel is not attended by any considerable loss in tensile strength as the results indicate. The successful removal of most of the scale from flame tube Inconel at room temperature by the NF solution is in no small part the result of the alkaline permanganate oxidizing pretreatment.

A full scale test of the NF solution was conducted at the Norton Air Force Base, San Bernardino, California. Inconel parts, flame tubes and transition liners were given the standard oxidizing pretreatment in hot alkaline potassium permanganate solution (TO-O2-IA-I3) and then pickled at room temperature for fifteen minutes in a 950 gallon tank filled with a solution 18% by weight in nitric acid and 2.3% by weight in ferric chloride hexahydrate. To facilitate the maintenance of proper control of the bath an analytical method was developed for the determination of the nitric acid content and the content of ferric chloride. The details of the analytical procedure are presented in Appendix XV.

Although the results of the test were generally quite satisfactory, a somewhat more reactive character was displayed by the solution in the large scale test than was apparent in the earlier laboratory studies. This enhanced activity was reflected both in a more thorough cleaning action and in more corrosion. During the one month test period the rejection rate for corrosion averaged 4.75% for inner combustion chambers and 0.00% for transition liners.

The NF solution failed to brighten the outer combustion chambers when they were immersed in the acid without an oxidizing pretreatment, whereas the cold ferric sulphate-hydrofluoric acid solution restored the "new look" to the outer chambers without any previous treatment, except degreasing and carbon removal. Because of the fact that the immersion of outer combustion tubes in the alkaline permanganate solution causes the expansion bellows to become contaminated with manganese dioxide these jet engine parts are not subjected to an oxidizing pretreatment at Norton Air Force Base. In view of the fact that the ferric sulphate-hydrofluoric acid solution performs acceptably in brightening these parts without an oxidizing pretreatment it will probably continue to be used in this particular application.

Diaphragm nozzles and turbine wheels are not subjected to any chemical descaling at Norton Air Force Base but are vapor blasted. For this reason the NF solution was not tested as a descaling medium for these jet engine components. In view of the success achieved in the laboratory descaling turbine blades with the NF solution following an oxidizing pretreatment in hot alkaline permanganate, the application of the NF solution to the cleaning of diaphragm nozzles and turbine wheels might be expected to give satisfactory results.

On the basis of these test results it can be stated that the NF solution is reasonably satisfactory in the descaling of Inconel parts such as flame tubes and transition liners. It is not apt to be employed in the brightening of stainless steel parts which for one reason or another cannot be given an oxidizing pretreatment in hot alkaline permanganate. Its value in cleaning up other parts such as diaphragm nozzles and turbine wheels has not been ascertained in full scale tests but appears promising on the basis of laboratory studies.

SECTION V - DESCALING MATERIALS AND METHODS DEVELOPED BY VENDORS

At the start of the Jet Engine Descaling Project at Northrop Aircraft, Inc. a questionnaire was composed and sent out to engine manufacturers, chemical companies interested in metal cleaning and to several military depots. The purpose of this questionnaire was to elicit information from industry and from the military concerning current practices in jet engine maintenance. The response to this questionnaire was somewhat disappointing. Three of the military establishments returned questionnaires containing considerable information concerning the frequency of certain types of jet engine cleaning yet no one professed to know anything about the nature of the scale deposits. Of the seventy-two commercial organizations contacted, only six ventured to propose any kind of treatment for the scaled surfaces. Most of the companies replied by returning the unanswered questionnaire with a letter explaining that they had no knowledge of the problem. Many of the firms who professed ignorance of the problem expressed willingness to conduct laboratory investigations if samples of scaled and smutted heat resistant alloy parts from jet engines were made available for experimentation. The questionnaire, the accompanying letter of introduction and the list of organizations to whom the questionnaire was sent is included in Appendix XVI.

Shortly after the returns from the questionnaire survey had been received and analyzed, the active participation of thirty-two companies in the jet engine program was invited by direct inquiry. A list of all of the organizations who were asked to participate in the program is presented in Appendix XVII. This list also indicates the extent to which each firm actually did participate in the program.

All of the vendors who were asked to participate in the program were supplied with at least one package of scaled Inconel specimens (about 20 pieces). As the work of each company progressed additional test material was supplied as it was required. Samples of descaled Inconel returned to this laboratory were given a visual examination and, in a few instances, microscopic examination. Each sample of descaled metal returned to the Northrop Laboratory for examination and appraisal was acknowledged by letter and the evaluation of the Northrop staff made known to the vendor. Vendors whose returned specimens were reasonably well cleaned and free from obvious corrosion were requested to send samples of their descaling formulations to the Northrop Laboratory for further evaluation. The vendors solutions were tested with samples selected and prepared according to the method outlined in Appendix VII. Inconel was the only test material. All vendors solutions

were tested with Inconel specimens which had had an oxidizing pretreatment in hot alkaline permanganate, and the descaled pieces were compared with pieces which had been similarly prepared and then treated with the NF solution.

Six vendors solutions were received which on a laboratory scale, at least, give descaling results comparable to the NF solution. These solutions are listed in Appendix XVII. None of these vendors solutions were given the extensive tensile and photomicrographic evaluation of the NF solution and the statement that serious corrosion is absent is based upon visual examination only. The final approval of these solutions as descaling agents for Inconel flame tubes and transition should await the outcome of full scale tests similar to the one conducted at Norton Air Force Base with the NF solution.

During the course of the Jet Engine Descaling Program at Northrop Aircraft, Inc., the Roto Finish Company of Kalamazoo Mich., became interested in the project and demonstrated how their mechanical tumbling process might be adapted to the cleaning of jet engine components. The first parts which Roto Finish cleaned were turbine blades and the most adequate description of the finished product is that the descaled blades resembled fine jewelry more than jet engine parts. They next cleaned several J-47 flame tubes with excellent results save for a slight denting of the lower edges of the tube. This mechanical damage can be attributed to the fact that the tubes were mounted in Roto Finish machines which were not designed to handle such large parts and could probably be easily eliminated by a slight modification of the equipment. Two J-47 flame tubes which were processed by Roto Finish are shown in Figure 9. The Roto Finish Company has been advised to consult with the Materials Laboratory, WADC, concerning such a development program since the extent of such an undertaking is beyond the scope of the present contract.

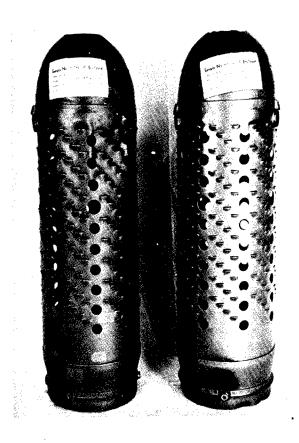


Figure 9. FLAME TUBES DESCALED BY ROTO FINISH PROCESS

The use of a high pressure stream of water containing optional amounts of graded abrasives to remove surface oxides is also a promising non-corrosive descaling procedure. A demonstration of this method applied to flame tube descaling was presented by representatives of the Hydro-Blast Company at the Oakland Foundry of the General Metals Corporation where a Hydro-Blast Machine is in operation. The results of the demonstration were quite good and the Hydro-Blast Company was advised by the author to confer directly with the Materials Laboratory, WADC, concerning the possibilities of a development contract.

Nine months of laboratory work and numerous conversations with Air Force personnel and jet engine manufacturers have convinced us that engine components made of Inconel or similar heat sensitized alloys should not be descaled. It is our first recommendation, therefore, that such parts not be subjected to descaling procedures, particularly with powerful acids above room temperature. The reasons we advance in support of this recommendation are:

- 1. The scale is a natural protective coating which prevents further oxidation. The repeated removal of scale followed by scale build-up in service is detrimental to the dimensional stability of the engine components, to say nothing of the strength of the base metal which may have suffered severe intergranular attack during descaling operations.
- 2. The scale is smooth and should not interfere with the proper flow of gases during service, provided, of course, that massive carbon deposits are removed. The surface of the scale, in fact, is often very much smoother than the base metal undermeath after the scale has been taken off.
- 3. The presence of the scale does not interfere with the inspection of the parts for flaws and imperfections, provided that adequate inspection techniques are employed.
- 4. Although the presence of the scale does interfere with the welding of cracks, it is easily removed from limited areas by mechanical means, or even by patch soaking with an appropriate acid. The immersion of the entire liner in an acid solution can produce greater damage than already exists in the form of small cracks around the vents and louvers.
- 5. It is unnecessary to use the formation of scale to check the operation of newly overhauled engines.

This recommendation does not apply to the outer combustion chambers, which are not made of Inconel, and, furthermore, never attain the high operating temperatures of the inner liners. We have not observed any damage to the outer combustion chambers from immersion in the hot ferric sulphate-hydrofluoric acid solution so damaging to the sensitized areas of the Inconel components.

The recommendation that Inconel parts not be descaled is made in the belief that all of the supporting statements (1) to (5) inclusive are correct. Should any of these statements be false, or at best only true under certain limiting circumstances, or if there are other important aspects of the jet engine cleaning problem which we have not considered, then the descaling of Inconel components may be necessary. If this is the case, our second recommendation is that the quality control standards be sufficiently relaxed so that flame tubes and transition liners can pass the cleaning inspection without having had all of the scale removed. It has been our repeated observation that the attempt to remove the last bit of scale by acid solvent methods inevitably results in such severe intergranular corrosion that the part is damaged beyond repair. The accomplishment of the removal of most of the scale with minimum damage to the base metal can be achieved with the NF solution or any one of the five vendors formulations listed in Appendix XIII of this report.

BIBLIOGRAPHY

- (1) Yamaguchi, S., Nakayama, T., and Katsurai, T.

 Electron Diffraction Studies on the Corrosion Resistance of Stainless Steel

 Journal of the Electrochemical Society of Japan. Vol. 95, No. 1 1949 pp21-24

 (English).
- (2) McCullough, H.M., Fontana, M.G., and Beck, F.H.

 Formation of Oxides on Some Stainless Steel at High Temperatures
 Trans. Am. Soc. Metals Preprint No. 4 1950
- (3) Ephraim, Fritz <u>Inorganic Chemistry</u> Fifth Edition Interscience Publishing Co., New York, 1949 p471
- (4) Latimer, Wendell M. and Hildebrand, Joel H. Reference Book of Inorganic Chemistry Revised Edition The Macmillan Publishing Co., New York, 1941 p474.
- (5) Ephraim, Fritz Inorganic Chemistry Fifth Edition Interscience Publishing Company, New York, 1949, p386.

APPENDIX I

JET ENGINE CLEANING AT TINKER AIR FORCE BASE, OKLAHOMA CITY, OKLAHOMA

In continuance of the survey of jet engine cleaning methods, a visit was made to Tinker Air Force Base, Oklahoma City, Oklahoma, on May 22nd and 23rd. The magnitude of the engine overhaul program at Tinker Field was far beyond the author's expectation. According to information presented with an engine display in the lobby, Tinker Field, the "Service Station of the Airforce" is scheduled to overhaul 462 B-29 Superfort engines and 381 turbo jet engines of all types per month. The latter figure, according to Col. Moore, the Commandant of the Engine Maintenance Division, will soon reach 400 jet engines per month. Over 25,000 civilian employees work at the base and the Engine Plant alone covers 35 acres. The overhaul facilities available in this tremendous installation, the presses, the shears, the foundry, the machine tools of every description, stagger the comprehension and the vast extent of floor space to be covered soon reduces even the most energetic visitor to impotent footsore fatigue.

This visit to Tinker Field has brought into sharp contrast the difference in opinions among AF Materiel officers concerning the proper objectives of jet engine cleaning. At Muroc, for example, the watchword is "Serviceability". Here the engine parts are degreased, stripped of paint and contaminants, and inspected before repair or replacement by the usual techniques. At Tinker Field, the engines are not only made as serviceable but even as bright and shiny as brand new equipment. It is now clear that Northrop Aircraft is expected to work towards the development of a descaling procedure which is now regarded as superfluous in some quarters.

Regardless of the relative merits of the two schools of thought regarding the extent to which heat resistant alloys must be cleaned, the operation at Tinker Field must certainly be credited with doing a beautiful job restoring the "new look" to old jet engines. In terms of time, and of money, however, the less vigorous methods in vogue at Muroc have much to be recommended. The published cost (on the Lobby Engine Display) of a Tinker Field overhaul of a J-47 engine is in the neighborhood of \$4,000. What this figure might be if the assembly line methods operated at design efficiency is difficult to appraise. The whole disassembly and cleaning operation is intended to reduce hand operations, yet the poor performance of the cleaning system requires a great deal of subsequent hand scrubbing and grit blasting.

The production of bright surfaces on heat resistant alloy parts is done with rather vigorous reagents including alkaline permanganate, ferrisol, and 25% nitric acid, and it is not accomplished without some sacrifice of base metal. The effect of these solutions upon the dimensions of the parts has never been evaluated, according to Colonel Moore. Inasmuch as very few assemblies have escaped some damage during service, the unbalance introduced by mechanical means; i.e., welding, grinding, replacement, etc., has so far obscured the erosive effects of chemical etching during cleaning. It is a matter of general concern, however, that the effect of repeated cleaning in these solutions upon the service life of the parts be evaluated because of the relative frequency of complete jet engine overhauls.

APPENDIX I

THE TINKER FIELD SYSTEM OF JET ENGINE OVERHAUL

The overhaul of jet engines at Tinker Field is planned for maximum efficiency and smoothness in the flow of engines and their components through the various operations of disassembly, cleaning, inspection, and reassembly lines; all centrifugual flow engines are placed in one line and all axial flow engines in the other.

As parts are removed, they are tagged to identify them with the engine to which they belong and are then suspended either individually or in baskets from the conveyor line which transports them from the disassembly area through a series of 9 tanks arranged in the form of the letter "S". In these tanks the parts are subjected to a sequence of cleaning operations and are supposed to emerge from the last tank, which is an air dryer, ready for inspection, or descaling and brightening in another series of cleaning reagents. Actually, the "S" system, as it is called, fails to do a complete job of grease, paint, dirt and carbon removal so that further hand cleaning or grit blasting is necessary to prepare many engine components for inspection. All engine components pass through the "S" system except certain small parts such as gears and bearings which are hand cleaned in special solvents and the compressor rotor assemblies of axial flow engines. The latter are subjected to vapor degreasing instead of the "S" system cleaning and are grit blasted whenever necessary.

The performance of the "S" system of washers is generally unsatisfactory from the viewpoint of efficiency and economy inasmuch as considerable hand scrubbing or grit blasting is necessary to prepare the parts for inspection after they have come off the conveyor line. The tough carbon deposits in the flame tubes may be softened somewhat but definitely are not removed. Paint is not removed uniformly from many parts, particularly magnesium castings, and grit blasting must always be resorted to for these engine components. The basic faults of the system are probably as much in its design as in the deficiencies of the solvents used. In regard to the solvent situation, it is worthy to note in passing that some of the solutions in the system are five years old. At the present time the settled sludge is removed periodically, but inasmuch as this method has allowed the system to operate in a perpetual overloaded condition, some modification is being considered. At the present time in the hope of increasing the cleaning efficiency, Mr. Stillman, the Chief Chemist, has recommended the purchase of an Oliver filter to separate the cleaning solutions from the continuously accumulating sludge of grease, dirt, carbon and paint. The author recommended the placement of a trichlorethylene vapor degreaser at the head of the line in order to remove the heavy burden of grease and oil from the now overloaded detergent solutions.

Heat resistant alloy components are sent to the descaling line after having passed through the "S" system, while the other parts are sent to inspection following the necessary hand cleanup or grit blasting. The descaling and brightening procedure begins in tank 10 which contains alkaline potassium permanganate and ends with tank 15 which is a water rinse. This procedure is producing parts which are as clean and bright as new but with somewhat smaller dimensions. The process is a bottleneck in the slow steps, particularly in the alkaline permanganate soak, and is quite costly in expensive and strategic material. The newly brightened components are examined for obvious flaws and are then inspected by the zyglo and magnaflux techniques. In the following section the defective parts are repaired or replaced and sent on for subassembly and balancing. After the subassembly and the balancing operations have been completed, the engines are completely assembled in the final department and are repackaged for shipment.

The following section of this report presents in tabular form and in the actual

sequence of operations certain pertinent facts concerning every step in the cleaning procedure now used at Tinker Field.

The chart presented on page 20 illustrates the general flow pattern of jet engine components from the receiving area to final assembly and packaging.

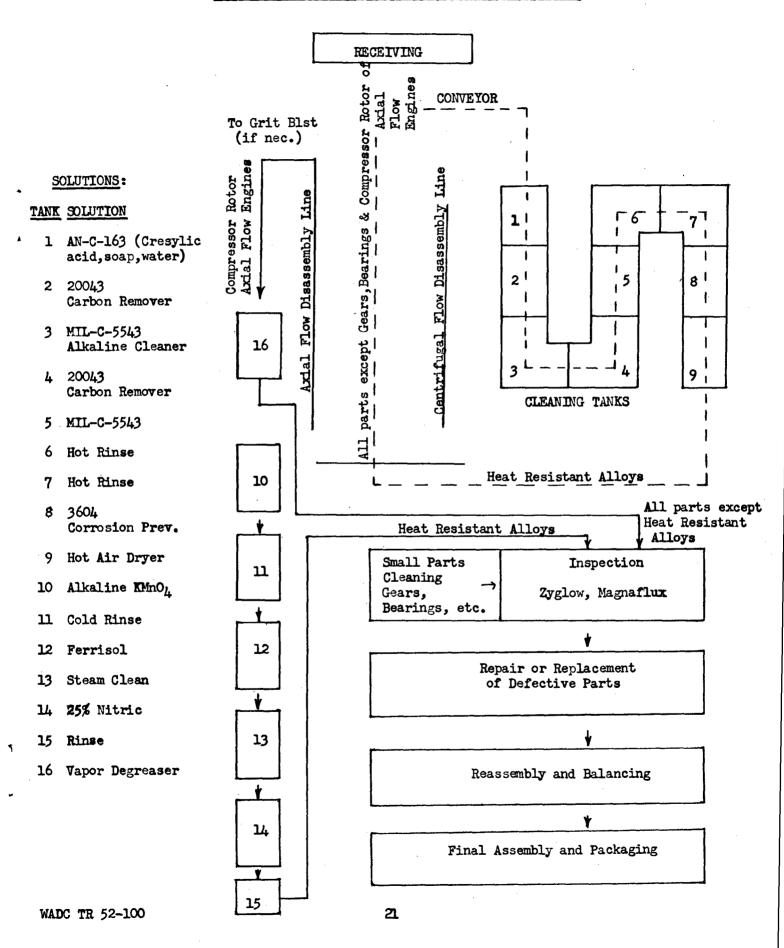
The jet engines, after having been uncoated in the receiving area, are placed in either of two disassembly lines depending upon engine type. Axial flow engines such as the G. E. J-47 are taken apart in the "north" line while centrifugal flow engines such as the Allison J-33 are disassembled in the south line.

The overhead monorail runs "countercurrent" to the disassembly lines and carries all parts except gears, bearings and the compressor rotor of axial flow engines into the S system of cleaning tanks.

Because the S system does a poor job of carbon and paint removal, a considerable amount of grit blasting and wire brushing must be done upon certain components after they have emerged from tank 9, the last station in the S system, before they can be released to inspection or sent ahead for further processing in the alkaline permanganate and ferrisol tanks.

The grit blast machines are not incorporated as integral parts of the jet engine cleaning system and parts which must be so treated have to be hauled a considerable distance through the plant. This unfortunate circumstance is not the result of poor planning but arises from the fact that the blasting equipment was originally installed to handle the components of piston engines and was located in the vicinity of the reciprocating engine line.

APPENDIX I FLOW CHART OF JET ENGINE OVERHAUL AT TINKER FIELD



APPENDIX I

THE CLEANING OPERATIONS AT TINKER FIELD "S" SYSTEM OF WASHERS

WADC TR 52-100

PERFORMANCE OF SOLUTION The maintenance of the necessary temperature and concentration of reactants is difficult. Carbon is definitely not removed.	This solution again does not remove the carbon but softens it somewhat.	Probably satisfactory. It is difficult to evaluate the performance of intermediate tanks because parts are not inspected until they have passed through the last tank.	This solution merely represents another attempt to remove carbon. It is not successful.		Satisfactory.
PURPOSE OF SOLUTION To degrease, to remove carbon and to strip paint.	To remove carbon, paint, grease and oil.	To remove grease and oil.	To remove carbon.	To remove grease and oil.	To rinse away traces of emulsified oil,
TEMP. OF BATH 150°F.	150°F.	not 150- - 160ºF. ch er are	1500F.	150- 160°F.	150- 160ºF.
INGREDIENTS AND CONC. Water 8% max Soap 30% max Cresols 62% min The soap shall be fatty acid 85% min; alkali, as K20 15% max.	Orthodichlorobenzene 53% min Cresylic acid 23-25% potassium oleate 10 t 12% Water 5% by volume Inhibitors 5% max.	The ingredients are not 150-specified by the gov- 1600 ernment. At Tinker Field the solids which are dissolved in water 4 oz. to the gallon are a mixture of: Sodium meta silicate Sodium carbonate Disodium phosphate			Water
SPECIFICATION NUMBER MIL-C-5546A	20043-A	MIL-C-5543	2004 3-a	MIL-C-5543	Hot rinse
NO. OF TANK 1	R	m	4	بر	2 8 9

Satisfactory.

detergents, etc. To prevent rusting.

Not specified

3604

₩

Hot Air Dryer

6

Satisfactory.

To dry parts.

APPENDIX I

DESCALING AND BRIGHTENING OF HEAT RESISTANT ALLOYS

PERFORMANCE OF SOLUTION	Solution is satisfactory except for the fact that its action is slow and it requires the frequent addition of permanganate. In a 6 mo, period last year 1300 pounds of permanganate were added. The settled sludge of manganese dioxide is removed and thrown away.	Satisfactory.	The ferrisol solution does the job but again the desired reactions are somewhat slow and attack upon the base metal always occurs, particularly near the extremes of the Fe ₃ (SO ₄) ₂ -HF ratio limits. Even if the ratio is maintained exactly at 4:1 some corrosion of heat sensitized parts is observed.	Satisfactory.	Satisfactory although possibly a bit rough on some heat sensitized alloys.
PURPOSE OF SOLUTION	To oxidize carbon and lower oxides of iron,chromium, nickel,etc.	To remove permang- anate, loose scale, etc.	To complete scale removal and to brighten metal.	To remove traces of ferrisol.	To passivate metal through the re- formation of an oxide film.
TEMP. OF SOLUTION	180-2000F.	Ambient	160-180°F.		Ambient
INGREDIENTS AND CONCENTRATION	Sodium hydroxide 16-20 oz/gal. 18 Sodium carbonate 16-20 oz/gal. Potassium permanganate 7-10 oz/gal. Water balance of solution	Cold rinse.	Ferric sulfate 6-10% by wt. hydrofluoric acid 1.5-2.5% by wt. ratio of Fe ₂ (SO ₄) to H F from 3.75 - 1 to 4.25 - 1	Steam tank.	25% nitric acid.
NO. OF TANK	91	ជ	. ដ	13	†T
WADC TR	52-100		23		

APPENDIX II

JET ENGINE CLEANING AT NORTON AIR FORCE BASE SAN BERNARDINO, CALIF.

The system of jet engine cleaning to be used at Norton Field is essentially the same as that now practiced at Tinker Field, Oklahoma City, Oklahoma. Certain modifications are being incorporated into the local system, however, which should effect considerable improvement in the efficiency of handling engines and materials and the general overall economy of the operation. The most notable improvements are:

- (1) A better overhead monorail system which allows engine parts to be sent through any desirable sequence of cleaning operations with a minimum amount of handling and dead time.
- (2) The use of superior cleaning tanks which provide for the use of a much wider variety of cleaning materials and which are equipped with filtration and distillation equipment to permit the removal of sludge and the recovery of solvents.
- (3) The incorporation of Vapor Blast and Grit Blast equipment as integral parts of the cleaning operation. At Norton Field, parts are transported to the "blast" equipment by the overhead monorail whereas at Tinker Field parts to be "blasted" must be taken off the monorail and carried by truck to a distant part of the building for the accomplishment of this operation.

The operation of the Norton Field system is illustrated schematically in the accompanying Flow Sheet.

- (a) As the engines are torn down on the disassembly lines, the parts are tagged and hung on the conveyor. All parts except the bearings and engine accessories are carried through the vapor degreaser. Bearings and accessories are sent through a solvent cleaning process and then to inspection and repair.
- (b) Following the vapor degreaser, another segregation of parts is effected. Those parts which need the treatment are transferred to the vapor blast line while those that do not, continue on through the 20043 soak tank.

Vapor Blast Equipment.

For the processing of parts which require vapor blasting, four units are available. These units which may be used on any part as required allow a variation in grit from 80 to 5000.

Soak Tank 20043.

The soak tank which is divided internally into three sections (1st and 3rd sections are 20043 solution while section 2 is a water spray) allows a part to remain in the carbon solvent for an hour while the monorail travels at the rate of 3 ft. per minute.

- (4) Parts out of the vapor blast system can be routed directly to any of the following operations:
 - (a) inspection and overhaul
 - (b) scale removal

APPENDIX II

JET ENGINE CLEANING AT NORTON AIR FORCE BASE SAN BERNARDINO, CALIF. (Con't.)

- (c) 20043 soak tank
- (d) industrial washer
- (5) The three stage industrial washer through which all parts passing through the 20043 soak tank are routed and through which some parts which have been vapor blasted are routed directly allows the use of any commercial detergent in the first two stages. The third stage is a water rinse.
- (6) The scale removal system is essentially the same as specified in TO O2B-1-13 and in use at Tinker Field. The use of a high pressure jet of cold water is more effective in knocking off the loosened scale following the ferrisol treatment than the steam cleaning used at Tinker Field. Incorporated also as a part of the scale removal equipment is a grit blast machine to assist in the removal of heavy carbon deposits or very stubborn scale. The actual etching of the metal is an important step in carbon removal; i.e., when the carbon cannot be removed from the scaled surface, the foundation of the deposit, scale and base metal must be dissolved to dissipate the contaminant.
- (7) Auxiliary Cleaning Equipment.

 To expedite the rapid rework of parts which have been returned from inspection for further cleaning, an auxiliary cleaning system has been installed close to the industrial washer. This system consists of 3 solvent spray booths and one vapor blast unit.

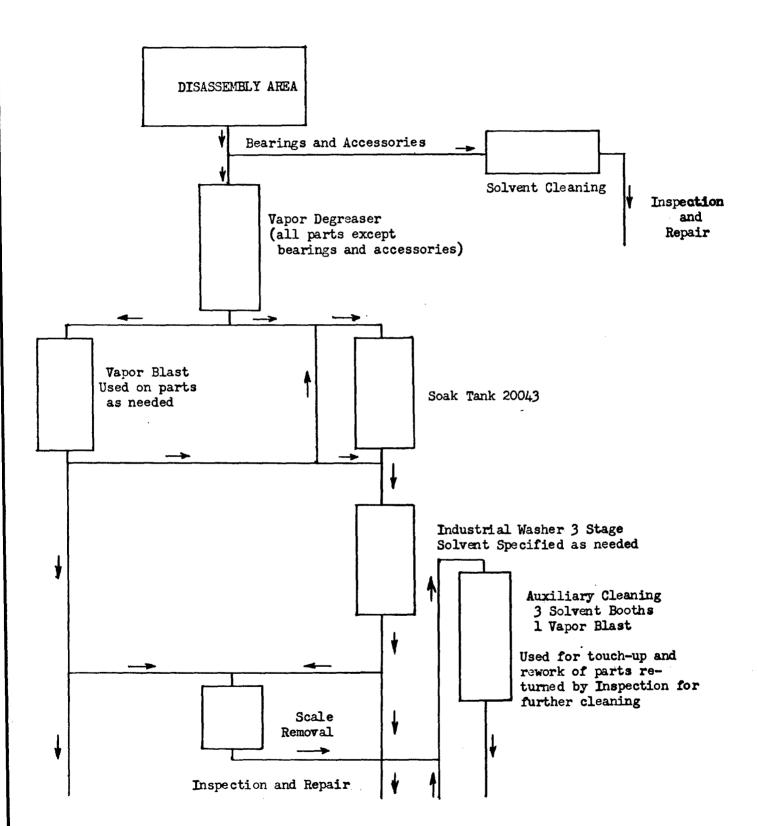
CONCLUSION: The jet engine cleaning system being built at Norton A.F. Base is undoubtedly the most advanced in the United States at the present time. Its limitations are not in the equipment available or in its disposition but are inherent in the materials currently available for engine cleaning.

25

APPENDIX II

JET ENGINE CLEANING SYSTEM, NORTON A.F. BASE, SAN BERNARDINO, CALIF.

FLOW SHEET



APPENDIX III

JET ENGINE CLEANING AT EDWARDS AIR FORCE BASE, MUROC, CALIFORNIA

On Tuesday, May 15th, the author was privileged to visit Edwards Air Force Base at Muroc, California and to inspect the Jet Engine cleaning and repair facilities. Despite the initial frustration of arriving at the Air Base before the complex machinery of security clearance had completed its operation, the trip was quite profitable. Major William A. Walker, Chief of Power Plant Operation, literally laid out the plush carpet and he was ably assisted in the task of demonstrating the problems involved in jet engine cleaning by Norman W. Shelby, Shop Superintendent. These remarkably fine gentlemen expressed great interest in the "project" and expressed their desire to assist the preliminary survey and the later experimental developments in every way possible.

This contact with the Power Plant Lab at Muroc should prove especially fruitful in view of the aims of the Edwards Air Force Base as regards Jet Engine Maintenance. It is the conviction of Major Walker that being a relatively simple machine, the jet engine should be capable of thorough overhaul at all Air Force Bases throught the world. Hence, cleaning methods should involve readily transportable equipment and easily available chemicals. He stated, in making his point clear, that the Edwards Base could not duplicate the cleaning procedure in operation at Oklahoma City nor did it ever intend to do so. At Edwards Field the mechanical reliability of the engine is the prime consideration, while the maintenance of glossy appearance, although desirable, is of secondary importance.

The engines whose "inner vitals" were exposed to view were no more numerous at Muroc than at the G. E. plant in Southgate. Fortunately, however, some of the engines being repaired at Muroc had undergone considerable service (200-300 hrs.) and some had suffered under shorter but more severe operation. In evidence were many examples of what happens to jet engine components in the field — shattered compressor blades — turbine wheels coated with oil varnish—burned off turbine buckets, etc. As a result of this visit the author's concept of the magnitude of the problem and his insight into the peculiar nature of many of its aspects are rapidly developing. In the following section are listed some of the principal engine components and the nature of the contamination observed on the respective parts.

ENGINE COMPONENT (S)	CONTAMINATION	REMARKS:	
Compressor	Oil and dirt on blades in fore section.	Can be wiped clean.	
	Oil varnish in "hot" aft section.	Oil varnish is difficult to remove. Vapor blasting is found to be effective. The compressor cannot be "dunked" as a unit. Hand cleaning of individual parts is necessary.	
Flame tubes	Carbon around fuel nozzles.	This carbon deposit is often quite difficult to remove.	
	Heat scale and stain on liners.	The scale and stain on the liners is not too objection-able in view of the relative frequent replacement of the parts.	

APPENDIX III

JET ENGINE CLEANING AT EDWARDS AIR FORCE BASE, MUROC, CALIFORNIA (Cont'd.)

ENGINE COMPONENT (3)	CONTAMINATION	REMARKS:
Transition liners.	Carbon and some heat scale.	These components are generally not too dirty.
Turbine wheel.	Cil varnish.	A nasty problem of considerable magnitude. Contamination removed by vapor blasting.
Turbine vanes and buckets.	Heat scale.	Not very thick deposit but quite adherent.
	Lead oxide.	Occurs when leaded fuel is used. Relatively heavy deposit is possible. Stain is very hard to remove, vapor blasting being the most efficient method found so far.
	Sprayed motal.	Comes from disintegrated compressor blades. Practically impossible to remove.

CLEANING PROCEDURE IN USE AT EDWARDS FIELD

The cleaning method which has been found sufficient to satisfy the requirements of the Power Plant Section at Edwards Field involves:

- (a) Degreasing in a trichlorethylene vapor degreaser.
- (b) Vapor blasting. (At the present time the vapor blasting operation is performed by Pacific Airmotive, Burbank. Maj. Walker expects that the new V-B Vapor Blasting Unit recently installed at the Base will be in operation within a short time.)
- (c) Solvent cleaning. The two solvent baths in use at Muroc were built under the direction of Turco Chem. Co. and utilize the solutions designated by that firm as Penetrol and Transpo, respectively.

APPENDIX IV

THERMAL ANALYSIS OF TURBO JET COMBUSTION ZONE

CHEMICAL ANALYSIS OF PRINCIPAL COMPONENTS	Ni 77%, Cr 14%, Fe 7%	Ni 77%, Cr 14%, Fe 7%	Co 63%, Cr 28%, Mo 6%, Ni 2%	Go 43%, Ni 20%, Cr 20%, Mo 4%, W Cb 4%, Fe 4%
METAL ALLOX	Inconel	Inconel	Vitallium	s-816
TRANSIENT MAX. TEMPERATURE	3000°F.	2400ºF.		
AVERAGE OPERATING TEMPERATURE	1850°F.	1700ºF.	1600°F.	1500ºF.
ENGINE PART	Flame Tube	Transition Liner	Nozzle Vanes	Turbine Buckets

1. The thermal data was compiled by the Power Plant Section of Northrop Aircraft, Inc.

APPENDIX V HEAT-RESISTANT ALLOYS FOR USE IN JET ENGINES

				TABL								
		77 75 3/	Di		lloys	A .	T m + A	\$1 TO	का वाहर	m		
Alloy	C	H E M.	Yn Mn	Cr Cr	O M P Ni	0 S :	ITIO Mo	N, PE W	Cb	Ti	N	Cu
			٨٠٠	ct on		30.00	Alloys					
Timken	0.10	0.75	1.00	16	25	base i	6				0.15	••
19-9DL	0.30	0.60	1.00	19	9	••	1.25	i	0.4	0.4	••	•••
Low-Carbon N155	0.15	0.40	1.50	20	2Ó	20	3	2	1	•••	0.15	•••
S590	0.45	0.50	1.00	20	20	20	4	4	4	••	••	•••
S816	0.45	0.50	0.75	20	20	45	4	4	4	••	••	••
eme	0.15	0.50	0.40	19	11	••	~	3.5	1.3	••	0.10	•••
CSA	10.40	0.30	4.00	18	5	••	1.5	1.5	0.6	••	••	•••
*G18B	0.40	1.00	0.80	13	13	10	2.0	2.5	3.0	••	••	•••
*Rex 78	0.08	••	••	14	18	••	4	••	••	0.6	••	4
	Î		۸۰۰	o Uo	rdena	h7 a A'	110					
	!		A g	e-na	ruena	DIG W	HOYS	Fe			Al	
Discalloy	0.05	0.70	0.70	13	25	••	3	55	••	1.8	0.20	
Inconel X	0.05	0.40	0.50	15	73	••	••	7.0	1.0	2.5	0.70	

				TABLE								
					Allo							
	<u>C</u>		ICAI	. C (0 S	ITI	ON, P				
Alloy	C	Si	Mn	Cr	Ni	Co	Mo	W	Cb	Ti	Al	
			٨٠٠	et on i	:+:0 1	Page	Alloys					N
Low-Carbon N155	0.15	0.4	1.5	20	20	20	3	2	1			0.15
5590	0.45	0.5	1.0	20	20	20	-	4	4	• •	• •	0.1)
5990 5816		0.5		20	20		4			• •	••	
	0.45	-	0.7			45	4	4	4	• •	• •	
Hastalloy B	0.05	0.7	0.7	••	65	••.	28	• •	• •	• •	••	_
							lloys					Fe
C42B	0.05	0.7	0.7	18	42	22	• •	• •	• •	2.2	0.2	14
Refractalloy 26	0.05	0.7	0.7	18	37	20	3	• •	• •	2.8	0.2	18
Inconel X	0.05	0.4	0.5	15	73	• •	• •	• •	1.0	2.5	0.7	7
Vimonic 80*	0.04	0.5	0.5	21	74	• •	• •	• •	• •	2.4	0.6	• •
Refractalloy 70	0.05	0.2	2.0	20	20	30	8	4	• •	• •	40	15
		•						•				Cu
Rex 78₩	0.08	• •	• •	14	18	• •	4	• •	• •	0.6	••	4
			P	recis	ion C	ast A	lloys					Fe
7itallium	0.20	0.6	1.0	25	3	61	••	5.5	• •	• •	• •	2
61	0.45	0.6	0.6	25	••	70	• •	5	• •	• •	• •	1
22-19	0.55	0.6	0.3	25	10	55	6	••	• •	••	• •	2 1 1
6059	0.40	0.8	0.2	25	35	35	5	• •	• •	• •	• •	1
K-40	0.50	0.7	0.6	25	10	55	••	7	• •	• •	• •	0.5
3816	0.45	0.5	0.7	20	20	45	L	į.	Ĺ	• •	• •	3

^{*} Analysis reported in U.S.A. work. ** English alloy.

APPENDIX V

HEAT-RESISTANT ALLOYS FOR USE IN JET ENGINES (Cont'd.)

			rials				
CHEM	CALC	OMPO	SITI	O N, PER	CENT		
C	Si	Mn	Cr	Ni	Co	W	Fe
0.15	0.50	1.0	13	75	• •	• •	9
0.05	2.0	1.8				••	52
0.20	0.6	1.0	25	3	61	5•5	2
	0.15 0.05	Flame Tu C H E M I C A L C C Si 0.15 0.50 0.05 2.0	CHEMICAL COMPO C Si Mn 0.15 0.50 1.0 0.05 2.0 1.8 0.20 0.6 1.0	Flame Tube Materials C H E M C A L C O M P O S T T	Flame Tube Materials C H E M I C A L C O M P O S I T I O N, PER C Si Mn Cr Ni 0.15 0.50 1.0 13 75 0.05 2.0 1.8 25 20 0.20 0.6 1.0 25 3	Flame Tube Materials C H E M I C A L C O M P O S I T I O N, PER CENT C Si Mn Cr Ni Co 0.15 0.50 1.0 13 75 0.05 2.0 1.8 25 20 0.20 0.6 1.0 25 3 61	Flame Tube Materials C H E M I C A L

REFERENCE: These Tables taken from the article, "Heat-Resistant Alloys for Use in Jet Propulsion Engines", by J. W. Freeman, E. E. Reynolds, and A. E. White, in JOURNAL OF THE AERONAUTICAL SCIENCES, December, 1947, Vol. 14, No. 12, pages 693-702 incl.

X-RAY DIFTRACTION ANALYSIS OF HEAT SCALE FORMED ON INCONEL LINER

Fezn	2.10 vs				
Fe ₂ 0 ₃	2.69 vs 2.51 s				
Cr203	2.67 s 2.47 s 2.03 w	1.67 vs 1.58 vw			
Nio	2.40 m 2.08 vs		1.474 m 1.258 m	1.203 w 1.042 vw	0.957 vw .933 vw .852 vw
a B	2.05 vs				
N.	2.04 vs	1.77 s	1.252 m	д 60°. г	.812 w
<u>د</u> ا	Intensity w w w m ms wvs	8 MAAA	: E E	MAA MAA	MAA
Lab. No. 111-23331	2 2 2 2 2 3 4 4 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5	1.67	1.475	1,200 1,068 1,042	0.952 0.952 .850 .812

s = strong, w = weak, m = medium, v = very

*This analysis was performed by the Metal Control Laboratories, 2735 E. Slauson, Huntington Park, California

32

SELECTION OF SAMPLES AND PREPARATION OF SPECIMENS

The following procedure was used in the selection and preparation of Inconel specimens for the testing of the most promising descaling solutions.

The dome of a J-47 flame tube and the lower flanged section were cut off with the aid of an oxy-acetylene torch and discarded. The more or less cylindrical sidewall was then cut down one side and flattened out, revealing very clearly the pattern of flame impingement by the variations in the color of the scale over the surface. The flattened side section was marked off with a colored pencil into sixteen vertical columns and three horizontal rows giving forty-eight pieces approximately 5" x 1.5". A serial number was engraved into the base metal of each piece. The number identified the flame tube from which each piece was cut and its exact location in the sidewall of the tube. A chart showed the number and location of each specimen and their assignment to specific sample groups. The engraved specimens were cut out of the sheet with a high speed saw, drilled near the upper end with a 1/4" bit to permit suspension in the test solutions, and then degreased with trichlorethylene. Massive carbon deposits were removed by scrubbing with soap and water.

Each specimen was identified upon the pertinent chart as belonging to one of three broad classifications of scale:

- (a) The very black and very refractory scale which generally develops near the top of the sidewall and which is most always accompanied by cracks around the louvers or massive buckling of the metal.
- (b) Dark blue scale which is not as refractory as the first type and which always presents a smooth surface.
- (c) Light scale which generally occurs near the lower end of the flame tube and which is frequently vari-colored.

Sample groups consisted of eighteen to twenty specimens. As a rule, three flame tubes would be prepared at the same time and each sample group would have specimens derived from all three tubes. A typical sample group would consist of five specimens covered with the heaviest black scale, eight specimens covered with the smooth dark blue scale, and six specimens covered with the light or vari-colored scale. All vendor formulations tested in the final phase of the program were evaluated with identical sample groups. It is believed that this system of sample selection helped to provide fair and objective evaluation of all promising descaling formulations.

APPENDIX VIII

ALKALINE AND NEUTRAL SOLVENTS TESTED AS INCOMEL DESCALING SOLUTIONS

SOLVENT	TEMPERATURE	EFFECT UPON THE SCALE
20% Sodium hydroxide	200°F•	No effect
28% Ammonium hydroxide	75°F.	No effect
10% Sodium cyanide	75°F.	No effect
10% Sodium fluoride	75°F.	No effect
10% Potassium thiocyanate	75°F.	No effect
20% Sodium hydrosulfite	200°F.	No effect
10% Sodium peroxide (aqueous suspension)	150° _F .	No effect
30% Hydrogen peroxide	75°F.	No effect
12% Potassium chlorate	200°F.	No effect
20% Sodium bromate	200°F.	No effect
20% Sodium iodate	200°F.	No effect
143 Potassium Perchlorate	200°F.	No effect
10% Potassium persulfate 5% Sodium Hydroxide 5% Sodium nitrate	75°F•	Slight loosening of the scale on a few specimens.
10% Sodium hypochlorite	75°F.	Severe pitting. Scale not removed.
10% Sodium hypobromite	75° _F .	Severe pitting. Scale not removed.
Molten sodium under toluene		No effect

^{1.} Unless otherwise specified in formulation, all solutions were aqueous.

APPENDIX IX

THE EFFECT OF PURE ACIDS IN VARIOUS CONCENTRATIONS

UPON INCONEL SCALE

ACID	CONCEN- TRATION	TEMP.	REACTION TIME	RESULT
Acetic	100%	160°F.	30 min.	No effect.
Acetic	25%	160°F.	30 min.	No effect.
Orthophosphoric	85%	160°F.	30 min.	No effect.
Ort hophosphoric	25%	160°F.	30 min.	No effect.
Hydrochloric	10%	160°F•	15 min.	Vigorous reaction but poor scale removal. Consider-able intergranular corrosion.
Sulfuric	10%	160°F.	30 min.	Some evolution of hydrogen but very little scale removal. Some intergranular corrosion.
Nitric	25%	160°F.	30 min.	Best of the straight acids, but the scale removal is generally far from complete. Very little intergranular attack

ACID FORMULATIONS CONTAINING REINFORCING AGENTS

HESULTS	No effect.	No effect.	This formulation was only partially effective against Inconel scale and was not considered sufficiently promising to merit further consideration.	Most of scale removed with no visible intergranular attack. The solution is unstable.	The scale was removed but with some evidence of intergranular attack.	The scale was removed but selective attack occurred at carburized areas as the solution aged.	The scale was removed but some inver- gramular corrosion occurred.	The scale was removed with very little evidence of intergranular attack. Nickel was deposited from the fresh solution upon the cleaned metal.
REACTION TIME	30 min.	30 min.	30 min.	20 min.	10 min.	10 min.	10 min.	15 min.
TEMP.	1400F.	140°F.	160- 180ºF.	70°F.	160- 1800F.	160- 180°F.	160- 160ºF.	160- 1300F.
TYPE OF REINFORC-ING ADDITIVE (s)	Oxidizing	Oxidizing	Oxidizing	Oxidizing	Complexing	Complexing and catalytic	Complexing and catalytic	Catalytic
ACID FORMULATION	Acetic acid 90% Lead tetracetate 10%	Orthophosphoric acid 44% Potassium permanganate 32%	Nitric acid 25% Lead droxide 5%	Nitric acid 16% Potassium chlorate 6%	Nitric acid 31% Hydrofluoric acid 1.3%	Nitric acid 31% Hydrofluoric acid 0.8% Zinc chloride 1%	Mitric acid 31% Mydrofluoric acid 1.3% Stannous chloride 0.8%	Nitric acid 5% Ferric chloride 5% Nickel chloride 5%

APPENDIX X

ACID FORMULATIONS CONTAINING REINFORCING ACENTS (Cont'd.)

RESULTS	The scale was removed with no visible intergranular corrosion.	The scale was removed with no visible intergranular corrosion.	No effect.	No effect.	Slight scale removal.	Practically all of the scale removed with no visible intergranular corrosion.
REACTION	20 min.	20 min.	20 min.	20 min.	20 min.	15 min.
TEMP	160- 180ºF.	160- 180ºF.	75°F.	200°F.	200°F.	70°F.
TYPE OF REINFORC- ING ADDITIVE (s)	Catalytic	Catalytic	Complexing and catalytic	Complexing	Complexing	Catalytic
ACID FORMULATION	Nitric acid 5% Ferric chloride 5% Cupric chloride 5%	Sodium bisulphate 5% Ferric chloride 5% Potassium nitrate 5%	Hydrochloric acid 0.4% Sodium nitrite 7.0%	Oxalic acid 1.8% Sodium oxalate 2.7%	Nitric acid 25% Calcium fluoride (satd.)	Nitric acid 25% Ferric chloride 2.3% (hexahydrate)

A. THE EFFECT OF VARIATION IN NITRIC ACID STRENGTH AND FERRIC CHLORIDE CONCENTRATION UPON THE DESCALING ACTIVITY OF THE NF ACID

CONCENTRATION OF NITRIC ACID	CONCENTRATION FERRIC CHLORIDE MOLES/LITER	DESCALING ACTIVITY
25%	0.001	All except heaviest scale removed.
25%	0.005	All except heaviest scale removed.
25%	0.010	All except heavisst scale removed.
25%	0.020	All except heaviest scale removed.
25%	0.040	All except heaviest scale removed.
25%	0.080	All except heaviest scale removed.
10%	0.001	Considerable medium scale not removed.
10%	0.010	Considerable medium scale not removed.
10%	0.100	Considerable medium scale not removed.

B. THE EFFECT OF SODIUM CHLORIDE AS AN ACTIVATOR

CONCENTRATION OF NITRIC ACID	CONCENTRATION OF SODIUM CHLORIDE MOLES/LITER	DESCALING ACTIVITY
25%	0.001	All except heaviest scale removed.
25%	0.003	All except heaviest scale removed.
25%	0.300	All except heaviest scale removed.

APPENDIX XII

THE EFFECT OF REPSO NF AND DIBUTYLTHIOUREA IN CERTAIN DESCALING ACIDS*

TOBASE	Inactive at first but improved with use.	No improvement - over 25% nitric acid.	Activity increased with use.	More active than straight NF acid at first but no different after aging period.	Inactive.	Scale removed with very little evidence of intergranular attack.
PICKLING	30 Min.	30 Min.	30 Min.	30 Min.	30 Min.	30 Min.
TEMP	75°F.	.75°F.	75ºF.	750F.	160ºF.	160°F.
CONC. OF DIEUTTIHIOURSA	0.00%	0.00%	%00.0	%00°0	205.0	0.50%
CONC. OF REPSO ME	0.00%	0.25%	%00.0	0.25%	0.25%	0.25%
ACID	Nitric Acid 25%	Nitric Acid 25%	NF Acid	NF Acid	Sulfuric Acid 10%	Hydrochloric Acid 20%

*All the Incorel test specimens were given a 1.5 hour pretreatment in hot alkaline potassium permanganate.

APPENDIX XIII

THE EFFECT OF VARIOUS OXIDIZING PREPTREATMENTS IN THE DESCALING OF STAINLESS STEEL WITH NF SOLUTION

OXIDIZING SOLUTION	TEMP.	REACTION TIME	RESULTS
Potassium permanganate Sodium hydroxide Sodium carbonate	0.5 molar 3.75 " 190°F. 1.50' "	2 hours	Very effective in promoting scale removal.
Potassium chlorate Sodium hydroxide	0.8 " 190°F. 3.75 "	2 hours	No effect.
Potassium perchlorate Sodium hydroxide	0.4 " 190°F. 3.75 "	2 hours	No effect.
Potassium persulfate Sodium hydroxide	0.4 " 70°F. 3.75 "	12 hours	Very effective in promoting scale removal.
Turco 3474A	190°F.	2 hours	No effect.

APPENDIX XIV

EFFECT OF VARIOUS DESCALING PROCEDURES UPON THE TENSILE STRENGTH OF INCONEL

ULTIMATE TENSIIE STHENGTH P.S.I.	93,250 93,310 93,250	93,920 93,720	94,870 93,140	93,340	93,870	88,9 10 89,970	89,970	64,530 57,950	75,910	80,520	83,440	90,500	000,06	007,68
PAST PICKLING TREATWENT	None None None	None None 1. hrs at 2000F.	4 hrs at 400°F.	None 4 hrs at 400°F.	-	None None	Kone	None None	hrs at	4 hrs at 400°F. None	None	None	None	None
PICKLING TREATMENT	None None	30 min in the NF solution at 1400F. 30 min in the NF solution at 1400F.	min in the NF solution at min in the NF solution at		min in the NF solution at	l'one None	None	30 min in the Mr solution at 140°F. 30 min in the NF solution at 140°F.	min in the NF solution at 1	30 min in the NF solution at 1400F.	min in the NF solution at 7	min in the NF solution at 7	min in the NF solution at min in the NF solution at	min in the NF solution
PRETREATMENT	None None	None None	None None $2 \text{ hrs Alk. } \mathbb{M}^{n}O_L$	hrs	2 hrs Alk. Kroo,	None None	None	None None	None	None	hrs Alk.	hrs Alk.	2 hrs Alk. $KMnO_{L}$	hrs
ORIGIN OF SPECIMEN	Virgin Sheet Virgin Sheet	Virgin Sheet Virgin Sheet	Virgin Sheet Virgin Sheet Virgin Sheet	Virgin Sheet	Virgin Shaet	Flame Tube	Flame Tube	Flame Tube	Flame Tube	Flame Tube	Flame Tube	Flame Tube	Flame Tube	Flame Tube

THE ANALYSIS OF THE NF SOLUTION

(2.55 normal nitric acid - 0.1 molar ferric chloride)

The molarity of ferric chloride must be known before the acid normality can be determined. The molarity of the ferric chloride can be estimated by means of a Zinnerman Reinhardt procedure which is presented below:

Pipet a 25 ml sample of the NF solution into a 250 ml beaker and add 2 ml of concentrated sulfuric acid. Carefully evaporate the solution to dryness to remove nitric acid and allow the residue to cool. To the cooled residue add not more than 5 ml of concentrated hydrochloric acid and 30 ml of distilled water. Heat the mixture to dissolve the solids, and, while it is near its boiling point, slowly add stannous chloride solution drop by drop until the yellow color of ferric iron is discharged. Add two drops in excess but no more. The color of the reduced solution will not be water white unless the sample has been taken from a new solution. If the sample has been taken from a bath which has been in use, the background color after the yellow ferric ion has been reduced may be a pale bluish green. After the reduction, add immediately 10 ml of 5% mercuric chloride to destroy excess stannous chloride. If the resulting precipitate is white and silky, pour the solution into a 600 ml beaker containing 300 ml of distilled water and 25 ml of Zimmerman Reinhardt titrating solution. Titrate immediately with 0.1 N KMnO4 until a faint pink color is obtained which persists for ten seconds. A dark precipitate indicates that too great an excess of stannous chloride has caused some of the mercuric chloride to be reduced to free mercury. Inasmuch as free mercury in a finely divided state reacts rapidly with permanganate, the results of the titration of a solution in which the reduction of mercuric chloride has gone too far will be in error.

The stannous chloride solution, which should be freshly prepared just before use, contains 150 g of iron free SnCl₂.2 H₂O in 1 liter of 1:2 hydrochloric acid.

One liter of the Zimmerman Reinhardt solution contains 70 g of MnSO $_{4}$.4 H $_{2}$ O, 125 ml of 85% phosphoric acid and 125 ml of concentrated sulfuric acid. The balance of the solution is distilled water.

The formula for calculating the molarity of the ferric chloride is:

 $M = \frac{M.N}{V}$

Where M = molarity of ferric chloride

M1 = milliliters of standardized permanganate

used in the titration

N = normality of the permanganate

V = volume of the sample

Once the molarity of ferric chloride has been determined it is possible to measure the acidity of the solution by a simple procedure.

THE ANALYSIS OF THE NF SOLUTION

(2.55 normal nitric acid - 0.1 molar ferric chloride)

Take a 10 ml sample of the acid and dilute to 200 ml with distilled water in a 400 ml beaker. Add three drops of phenolphthalem indicator and titrate with a standard 1 N sodium hydroxide until the pink color of phenolphthalem can be observed in the supernatant liquid above the precipitated ferric hydroxide. The normality of the solution can be calculated by the following formula:

$$N = \underbrace{M1.N}_{V} - 3M$$

Where N = Normality of the acid.

M1 = Volume in milliliters of standard caustic solution used in titrating to the phenolph-thalem endpoint.

M = Molarity of ferric chloride.

V = Volume of the sample.

APPENDTX XVT

QUESTIONNAIRE SURVEY INTRODUCTORY LETTER

In reply refer to: 2000-1533

TEP:KFF:dml

NORTHROP AIRCRAFT, INC. Hawthorne, Calif.

7 May 1951

TO: (See distribution list pages 46 to 48)

INTRODUCTION

The overhaul, repair, servicing and inspection of jet engines and turbo-jet engines requires removal of heat scale and smut from hot sections in order to accomplish these operations with greatest facility and efficiency. Military airplanes in service are often isolated from maintenance installations which afford elaborate processing equipment and procedures.

The USAF has designated Northrop Aircraft, Inc. to conduct a program aimed at establishing a simplified procedure for cleaning jet engines and turbojet engines in order to overcome the shortcomings of present practices under the circumstances noted above.

Whereas the primary goal of this program involves simplicity, it also is necessary to select processes and chemicals which demonstrate optimum economy and effectivemeness. With these factors in mind the Northrop Company turns to you for counsel as a contributor to the successful accomplishment of the USAF program.

Will you please complete the attached questionnaire and return by May 15, 1951, to Project No. 77, Attn.: Keith F. Finlay, Northrop Aircraft, Inc., Hawthorne, California.

Very truly yours,

NORTHROP AIRCRAFT, INC.

T. E. PIPER Chief Materials and Process Engineer

QUESTIONNAIRE

- I. To what extent do you dismantle jet engines for overhaul, servicing and inspection?
 - a. For routine maintenance.
 - b. For periodic overhaul.
- II. At what frequency (according to engine model and airplane model) is cleaning necessary for overhaul, repair, servicing and inspection?
- III. What method do you recommend for removing heat scale and smut from jet and turbo-jet engines?

Please note all significant aspects of recommended procedure, such as:

- a. Composition of cleaning solutions.
- b. Number of stages of baths and their chemical composition.
- c. Optimum operating temperatures.
- d. Maximum and minimum permissible operating temperatures.
- e. Sequence in which specific engine components are processed, with respect to their progression from bath to bath.
- f. Average time for exposure of specific engine components to each bath.
- IV. How long in average usage conditions does each bath operate effectively before renewal is necessary?
- V. Do any of the baths require periodic admixture of inhibitors or fortifiers?
- VI. If the answer to V. is Yes, what additives are required and how often must they be added to the various solutions?
- VII. What are the health hazards involved in the use of these solutions?
- VIII. What are the physical characteristics of the deposits encountered in various engine components? (i.e. hard, soft, porous, lamilar, etc.)
 - IX. If you have typical chemical analyses of scale and smut found in various areas of the engines, will you please note according to component and location.
 - X. What deterioration of dimensions, weight and balance have you observed as a result of your present cleaning procedure?
 - XI. From your experience with jet engine cleaning, what are the most needed improvements in cleaning methods?
- XII. At what frequency (according to engine and airplane models and specific components) is cleaning necessary for overhaul, repair, servicing and inspection?

- XIV. Has hydrogen embrittlement induced by cleaning been a problem? If so, under what conditions did it occur and how was it remedied?
- XV. How is rusting and oxidation inhibited on parts subsequent to cleaning?

NAME	
COMPANY	
ADDRESS	

DISTRIBUTION OF JET ENGINE QUESTIONNAIRE

AIR FORCE AND NAVAL REPAIR BASES

Edwards Air Force Base* Muroc, California

Eglin Air Force Base* Valparaiso, Florida

George Air Force Base Victorsville, California

Hamilton Air Force Base Hamilton, California

Naval Air Station Alameda, California

Naval Air Station Bethesda, Maryland

Norton Air Force Base San Bernardino, California

Tinker Air Force Base*
Oklahoma City, Oklahoma

PRIVATE INDUSTRY

(Manufacturers of aircraft power plants)

Aerojet Engineering Corp. Azusa, California

Allison Co.*
Div. General Motors
Indianapolis, Indiana

Curtis-Wright Corp.
Wood-Ridge, New Jersey

Ex-Cell-o Corp.
Detroit, Michigan

Fairchild Engine and Airplane Corp. Hagerstown, Maryland

Solar Aircraft Co.*
San Diego, Calif.

General Electric Co.★ West Lynn, Mass.

McGregor and Simler Mfg. Co. Birmingham, Mich.

Marquardt Aircraft Co. Van Nuys, Calif.

Pratt and Whitney Aircraft E. Hartford, Conn.

Reaction Motors, Inc. Rockaway, New Jersey

Westinghouse Electric Corp. Pittsburgh, Pa.

*Reply to questionnaire contained information pertinent to jet engine cleaning.

DISTRIBUTION OF JET ENGINE QUESTIONNAIRE

CHEMICAL MANUFACTURERS AND MANUFACTURERS OF METAL CLEANING COMPOUNDS

Aeroil Products Co. Inc. New York, New York

L. B. Allen Co. Chicago, Illinois

American Chemical Paint Co.*
Ambler, Pennsylvania

Apex Chemical Mfg. Corp. Detroit, Michigan

Apothecaries Hall Co. Waterbury, Conn.

Chemicals, Inc. Joliet, Ill.

Clifton Chemical Corp. New York, New York

D. C. Cooper Co. Chicago, Illinois

Dearborn Chemical Co. Chicago, Illinois

Diversey Corp. Chicago, Ill.

Du-Lite Chemical Corp. Middletown, Conn.

E. I. du Pont de Nemours and Co. Wilmington, Delaware

Enthone, Inc. New Haven, Conn.

Etsol Synthetic Prod. Co. Detroit, Michigan

Excelo Mfg. Co. Alhambra, Calif.

Fischer Industries, Inc. Cincinnati, Ohio

James Good Company Philadelphia, Pa. Hanson-Van Winkle-Manning Matawan, New Jersey

Heatbath Corp.*
Springfield, Mass.

Holcomb Mfg. Co. Indianapolis, Ind.

R. M. Hollingshead Corp.* Camden, N. J.

E. F. Houghton Co. Philadelphia, Pa.

Kelite Products, Inc. Los Angeles, Calif.

H. Kohnstamm and Co. New York, N. Y.

L'Hommedieu Co. Chicago, Ill.

L and R Mfg. Co. Arlington, N. J.

MacDermid, Inc.* Waterbury, Conn.

Magnus Chemical Co. Garwood, N. J.

Maritime Chemical and Repair Co. Brooklyn, N. Y.

Midwestern Mfg. Corp. New York, N. Y.

J. C. Muller Co. Grand Rapids, Mich.

Mitchell-Bradford Chemical Co. Stratford, Conn.

Munning and Munning, Inc. Newark, N. J.

Nielson Chemical Co. Detroit, Michigan

DISTRIBUTION OF JET ENGINE QUESTIONNAIRE

CHEMICAL MANUFACTURERS AND MANUFACTURERS OF METAL CLEANING COMPOUNDS

Nopco Chemical Co. Inc. Harrison, N. J.

Northwest Chemical Co. Detroit 4, Michigan

Nu Steel Co. Chicago, Ill.

Oakite Products, Inc. New York, N. Y.

Octagon Process, Inc. Brooklyn, N. Y.

Optimus Detergents Co. Matawan, N. J.

Park Chemical Co. Detroit, Mich.

Parker Rust Proof Co. Detroit, Mich.

Peerless Chemical Co. Detroit, Mich.

Philadelphia Quartz Co. Philadelphia, Pa.

Philadelphia Rust-Proof Co. Philadelphia, Pa.

Phillips Mfg. Co. Chicago, Ill.

Practical Products Co. Minneapolis, Minn.

Quaker Chemical Prod. Corp. Conshohocken, Pa.

Rex Oil and Chemical Co. Cleveland, Ohio.

Rumford Div. - Heyden Chem. Corp. Rumford, R. I.

Rustain Products, Inc. New York, N. Y.

E. B. Snyder Laboratories Philadelphia, Pa.

Spazier Soap and Chemical Co. Santa Monica, Calif.

W. L. Spencer Co. Milwaukee, Wis.

Stevens-Wiley Co. Inc. Philadelphia, Pa.

Sully Engineering, Ltd. Los Angeles, Calif.

Tesco Chemicals, Inc. Atlanta, Ga.

Turco Products, Inc. Los Angeles, Calif.

United States Steel Supply Co. Chicago, Ill.

Victor Chemical Works Alhambra, Calif.

Western Reserve Laboratories* Cleveland, Ohio

Wyandotte Chemicals Corp. Wyandotte, Mich.

METHODS OF SCALE REMOVAL RECOMMENDED IN REPLY TO QUESTIONNAIRE

COMPANY

TREATMENT RECOMMENDED

American Chemical Paint Co. Ambler, Pennsylvania Caustic-detergent treatment for removal of lead oxide. Heat scale should not be removed because the scale affords protection of the base metal against further oxidation, and because currently used methods of scale removal cause a loss of base metal and introduce the possibility of hydrogen embrittlement.

Western Reserve Laboratories Cleveland, Ohio

Manganesed Phopholene #7.

Heatbath Corporation Springfield, Mass.

Oxidizing molton salt bath followed by acid pickle.

Solar Aircraft Co. San Diego, California Oxidizing molten salt bath followed by acid pickle.

R. M. Hollingshead Corp. Camden, N. J.

Acid cleaner-identity undisclosed.

MacDermid, Inc. Waterbury, Conn.

Mechanical cleaning such as the Roto Finish Process. This company has tried many different solutions, both alkaline and acid, but has not been successful in their attempts to remove heat scale and smut from jet engine components by such means.

LETTER OF SOLICITATION

NORTHROP AIRCRAFT, INC.
Hawthorne, Calif.
25 June 1951

In reply refer to: 2000-2138

GMB:H:ams

Gentlemen:

The U.S.A.F. has designated Northrop Aircraft, Inc. to conduct a program aimed at improving present descaling procedures for jet engines. In an attempt to establish the scope of the problem, we prepared a questionnaire which sought information concorning present jet engine cleaning methods, and we circulated this questionnaire among jet engine producers, metal cleaning establishments, and chemical manufacturers. We have been somewhat surprised to find in reviewing the returned questionnaires that very few companies whom we have considered as probable experts in the field knew anything about it. Because we have lately had several opportunities to observe jet engine overhaul operations in the field, we are now in the position to provide more specific information to anyone who may be interested in the problem. Enclosed with this letter are copies of two reports made by Dr. Bryan of our laboratories after he had visited Edwards Field at Muroc, California and Tinker Field at Oklahoma City, Oklahoma. We believe that these memoranda present a fairly complete picture of the situation, particularly in regard to the difference in opinion among Air Force men concerning the necessity of removing oxide scale from heat resistant alloys.

THE PROBLEM

Stated simply, this is the problem:

"To remove heat scale and smut from jet and turbo jet engines."

Unfortunately, we have not found much useful information in the chemical literature concerning the constitution, structure, and properties of the heat scale which develops on stainless steel and other high temperature alloys during jet engine operation. It does not seem unreasonable to assume, however, that this scale is a mixture or molecular compound of the oxides of some or of all the metals which compose the alloy. Some of the present methods of removing this scale indicate its oxide character. Considering the molten salt processes, one variation depends upon the oxidation of the scale to higher, more soluble "valances" by sodium nitrate, while another relies upon the reduction of the scale by sodium hydride. The molten salt method, incidentally, is frowned upon by the Air Force because of the difficulty which is experienced in removing the salt from the many crevices in complex assemblies and because of the possibility that unremoved salt may promote subsequent corrosion. The present "wet" method of scale removal used by the Air Force starts with a preliminary oxidation in alkaline potassium permanganate at 150-160°F. and is completed, following an intermediate rinse, by a ferric sulfate-hydrofluoric acid solution, "ferrisol", at 160°F. The subsequent rinse in 25% nitric acid is a metal passivation process and is not an integral part of the scale removal. The principal objections to this process are that the ferrisol solution is too corrosive, especially on heat sensitized alloys, that the preliminary oxidation in alkaline permanganate is too slow, and that the oxidizing ingredient, potassium permanganate, is expensive and in critical supply.

LETTER OF SOLICITATION

We are of the opinion that any satisfactory method of scale removal will probably involve either an exidation or a reduction process, or possibly a combination of the two. For the sake of preventing intergranular corrosion and hydrogen embrittlement, a neutral or alkaline solution appears superior to a low pH medium. Complexing or "chelating" reagents may be necessary to increase the driving force of the solubilizing reaction.

If the development of a new non-corrosive method of high temp alloy descaling offers reasonable commercial possibilities to your organization, Northrop Aircraft, Inc. invites you to participate in this program. We will endeavor to supply you with samples of heat scaled "stainless" metals as they are released to us by the U.S.A.F. and to make available to you the assistance of the Northrop Aircraft, Inc. Process Engineers who are assigned to this project. Naturally, any new processes which are "dreamed up" in your laboratory belong to you because you will have assumed no contractural obligations with us or the U.S.A.F. All technical correspondence is a matter of the highest confidence. In fact, as far as formulations are concerned, we have an interest in knowing only their general characters. It's the results that count!

We extend our sincere thanks for the interest you may have already expressed in this project and we hope that your participation may help its successful and speedy conclusion.

Very truly yours,

T. E. PIPER Chief Materials and Process Engineer

Encl.

APPENDIX XVII

VENDORS PARTICILATING IN JET ENGINE PROGRAM

MATERIAIS SUBMITTED SUBMITTED (Vendor's UATION Designation) REMARKS	Ambient Par. 10-X Heavy scale not removed.	None	None	None	None	950°F. Com. None Perfect scale removal	None	None	None	None	None	None
- CLEANING OPERATING METHOD TEMP.	Hild Acid Amb					Molten salt 9 Mild acid						
PIECES NE- TURNED	Yes	No	o N	No	No	Yes	No	No	No	No	No	No
PARTIC- IPATING	Yes*	.v.Yes*	¥se _Y	Yes*	YeeY	Yos*	*se7	Zes*	Yes*	¥es*	Yes*	Yes*
		American Chemical Paint Co.Yes* Ambler, Pa.	Conn.	Cee Doe Chemical Co. Los Angelas, Calif.	Diversey Corp. South Gate, Calif.	E.I. duPont de Nemoures Wilmington, Del.	Enthone, Inc. New Haven, Conn.	Hanson-Van Winkle-Manning Matawan, N. J.	Heatbath Corp. Springfield, Mass.	R.M. Hollingshead Corp. Camden, N. J.	E. F. Houghton Co. Philadelphia, Pa.	International Nickel Co.

APPENDIX XVII

VENDORS PARTICIPATING IN JET ENGINE PROGRAM

WENDOR Kelite Prod. Inc. Los Angeles, Calif. H. Kohnstamm and Co. New York, N. Y.	PARTIC- IPATING Yes* Yes*	PIECES RETTURNED NO NO Yes	CLEANING O METHOD METHOD	OPERATING TEMP.	EVAL- UATION Com.	MATERIALS SUBMITTED (Vendor's Designation) None None	REMARKS Excellent scale removal.
Detroit, Mich. Magnus Chem. Co. Garwood, N. J. Nielson Chemical Co.	Yes *	on o	Mild acid			None None	
Northwest Chemical Co. Detroit 4, Mich. NuSteel Co. Chicago, Ill.	Yes# Yes#	No No				None None	
Oakite Prod. Inc. New York, N. Y. Octagon Process Inc. Brooklyn, N. Y.	Yes*	Yes No	Mild acid	160°F.	Par.	Composition None	Heavy scale not removed.
Optimus Detergent Co. Matawan, N. J.	Yes*	No				None	
Park Chemical Go. Detroit, Mich.	Yes*	No					
Refined Prod. Corp. Lyndhurst, N. J.	Yes*	No				None	

APPENDIX XVII

,

VENDORS PARTICIPATING IN JET ENGINE PROGRAM

VENDOR	PARTIC- IPATING	PIECES RE- TURNED	CLEANING O METHOD	OPERATING TEMP.	EVAL- UATION	MATERIALS SUBMITTED (Vendor's Designation)	FEEWARKS
Roto Finish Kalamazoo, Mich.	Yes*	Ĭes	Tumbling with mild acid	Ambient	Com.	None	Excellent scale removal.
Ryan Aircraft Co. San Diego, Calif.	¥es.	Yes	Molten salt strong acid	900°F.	Com.	None	Heavy scale removed. I.C.
Solar Aircraft Co. San Diego, Calif.	Yes*	Yes	Molten salt mild acid	900°F.	Com.	None	Perfect scale removal.
Solventol Chem. Prod. Detroit, Mich.	Yes*	No				None	
Turco Prod. Inc. Los Angeles, Calif.	Yes*	Yes	Mild acid	165°F.	Par.	3474	Heavy scale not removed.
Western Reserve Labs. Cleveland, Ohio	Yes*	Kes.	Mild acid	165°F.	Par.	M.P. #3	Heavy scale not removed.
Wyandotte Chem. Corp. Wyandotte, Mich.	Yes*	Yes	Mild acid	Ambient	Par.	Nitro- MF Acid	Heavy scale not removed.
Yates Products Glendale, Calif.	*ee*	No				None	

^{*} Test scaled pieces supplied.
Par. - partial scale removal.
Com. - complete scale removal.
I.C. - intergramular corrosion.